RESEARCH ARTICLE OPEN

Feasibility Study of Synthesis of Nanostructured Aluminum Nitride Through Sol-Gel Route

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I. INTRODUCTION

Aluminum nitride is a wide band gap semiconductor (6.2 eV) material with high thermal conductivity, electrical resistivity, break down voltage and low thermal expansion coefficient [1-2]. The main applications of aluminum nitride are in optoelectronics, as substrate and packaging material in microelectronic devices, as a heat sink etc. It is also used as SAW sensors owing to its piezoelectronic properties[3].

Aluminum nitride can be produced by different synthesis methods like – combustion synthesis, RF plasma sputtering, metal-organic chemical vapor deposition, pulse laser ablation etc.[4] – [10]. However direct nitridation of aluminum and carbothermal reduction followed by nitridation (CTRN) of alumina are conventional techniques for the synthesis of aluminum nitride. Aluminum nitride powder with uniform particle size can be produced by CTRN process and this improves the sinterability of the material. Moreover, AlN produced by this method is also very pure and stable against humidity. But the main problem with CTRN process is that it requires very high temperature of 1400 - 1800^0C and considerably long reaction time. AlN has been prepared form Al_2O_3 and carbon by Forslund and Zheng at 1600^0C after 4 hours of heat treatment in an elevated nitrogen pressure of 0.5 MPa [11]. Spherical Al_2O_3 particles are reduced and nitrided to spherical AlN by a gas mixture of NH_3 and C_3H_8 at 1500^0C [12]. Here in this paper a we report a method of synthesis of aluminum nitride by the carbothermal reduction followed by nitridation of alumina gel.

II. EXPERIMENTAL TECHNIQUES

Aluminum isopropoxide (98%) and dextrose are used as the precursor of aluminum and carbon respectively. Aluminum iso propoxide and dextrose are mixed in 100 ml of water and hydrolyzed for 2 hours at 80^0C. After obtaining the white precipitate of bhoemite 0.016 moles of conc. nitric acid is added in the solution and peptized for 46 hours at 80^0C under constant stirring. The resultant solution is left in a beaker for 7 days to get alumina gel. The molar composition of the alumina gel thus prepared is aluminum isopropoxide: water is 1: 100 containing 200% of excess stoichiometric dextrose required for the carbothermal reduction of alumina. Dried gel samples are heat treated in a PID controlled furnace. The temperature of furnace was raised to 1200^0 - 1350^0C and held at that temperature for 3 hours. After attaining the heat treatment temperature one gel sample is placed in a charcoal boat and pushed at the hot zone of the furnace. The heat treatment is carried out in nitrogen atmosphere. The heat treated samples are ground in an agate mortar and passed through 300 mesh sieve. XRD patterns of the powdered samples were recorded using Rigaku Ultima III X-Ray deffractometer with Cu Kα radiation in a continuous scan mode from 20^0 - 80^0. The powder sample is placed on a conducting carbon tape and FESEM (Hitachi made, Model no. S - 4800) is used to observe the morphological details of the phases present. FTIR is carried out to using Shimadzu spectrometer (model: Prestige 21). A small amount of ground sample is mixed with KBr and a pellet is formed. The spectrum is obtained over 4000 – 450 cm^{-1} at room temperature. The spectrum of pure KBr is used as the back ground.

III. RESULTS & DISCUSSION

Fig. 1(a) – 1(c) is the XRD pattern of the sample heat treated at 1200, 1300 and 1350^0C for 3 hours in argon atmosphere with prior repeated evacuation and purging with nitrogen gas. Alumina is partially converted to aluminum nitride and the XRD pattern shows presence of both alumina and aluminum nitride peaks when heat treated at 1200^0C. Complete conversion of alumina to aluminum nitride is achieved both at 1300 and 1350^0C and only aluminum nitride peaks are observed in the corresponding XRD pattern.

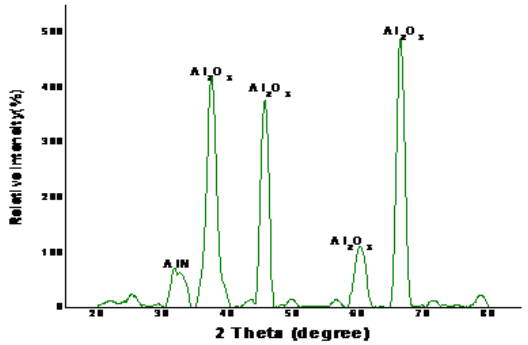
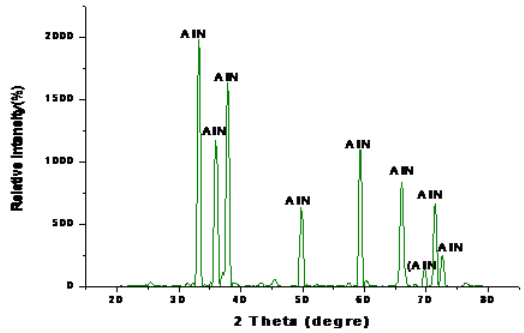


Fig. 1(a): XRD pattern of the sample heat treated at 1200^0C for 3 hours in nitrogen atmosphere.



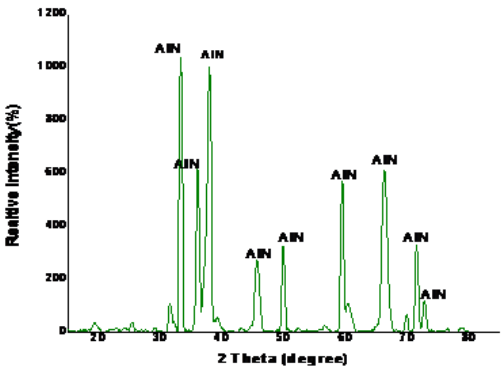


Fig. 1(b): XRD pattern of the sample heat treated at 1300^0C for 3 hours in nitrogen atmosphere.

Fig. 1(c): XRD pattern of the sample heat treated at 1350^0C for 3 hours in nitrogen atmosphere.

Fig. 2 is the FESEM image of the sample heat treated at 1350^0C for 3 hours in nitrogen atmosphere with prior evacuation followed by purging with nitrogen gas. The image shows agglomerated particles of aluminum nitride (confirmed by XRD). The size of the nanoparticles is in the range of 20- 50nm.

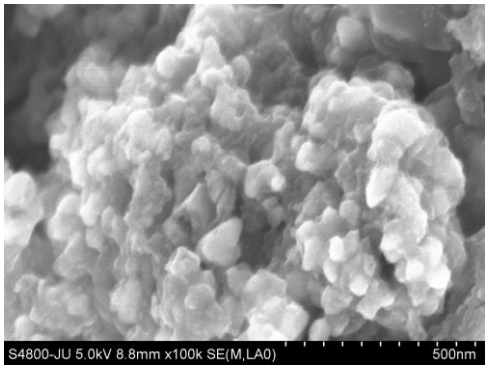


Fig. 2: SEM micro image of the sample heat treated at 1350^0C for 3 hours in nitrogen atmosphere.

Observations based on FESEM and XRD analyses are further confirmed by FTIR spectroscopic analysis. Fig. 3 shows the IR spectrum of the sample with 200% excess dextrose gel heat treated at 1350^0C for 3 hours under a constant flow of nitrogen with prior evacuation of the furnace followed by purging with nitrogen gas. The IR strong absorption band at 705 cm^{-1} correspond to the characteristics absorption bands of Al – N [13] and this confirms the formation of AlN. The weak band present at 3423 cm^{-1} corresponds to H – O bond.

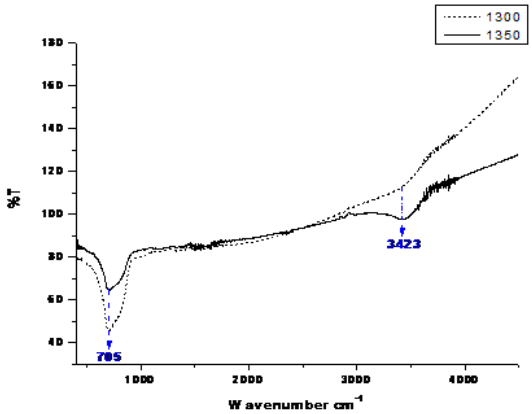


Fig. 3: FTIR spectrum of the sample heat treated at 1350^0C for 3 hours in nitrogen atmosphere.

IV. CONCLUSION

- Aluminum nitride can be synthesized by the heat treatment of alumina gel in nitrogen atmosphere at a minimum temperature of 1300^0C.
- Morphology of aluminum nitride produced is spherical with a particle size of 20 – 50nm.

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